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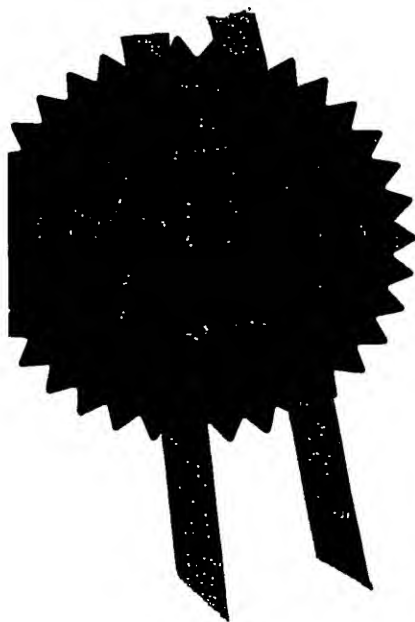
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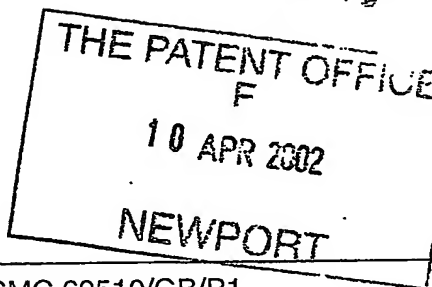
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1. Your reference

SMC 60510/GB/P1

2. Patent application number

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3. Full name, address and postcode of the or of each applicant (underline all surnames)

Avecia Limited
Hexagon House
Blackley, Manchester
M9 8ZS

Patents ADP number (if you know it)

7764137001

If the applicant is a corporate body, give the country/state of its incorporation

United Kingdom

4. Title of the invention

PROCESS FOR CHEMICALLY PRODUCED
TONER

5. Name of your agent (if you have one)

GILES, David Eric

"Address for service" in the United Kingdom to which all correspondence should be sent (including the postcode)

Avecia Limited
PO Box 42, Hexagon House
Blackley, Manchester
M9 8ZS

Patents ADP number (if you know it)

6660914002

7764137001

6. If you are declaring priority from one or more earlier patent applications, give the country and the date of filing of the or of each of these earlier applications and (if you know it) the or each application number

Country

Priority application number
(if you know it)

Date of filing
(day / month / year)

7. If this application is divided or otherwise derived from an earlier UK application, give the number and the filing date of the earlier application

Number of earlier application

Date of filing
(day / month / year)

8. Is a statement of inventorship and of right to grant of a patent required in support of this request? (Answer 'Yes' if:

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 - b) there is an inventor who is not named as an applicant, or
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Description

Claim(s)

Abstract

Drawing(s)

12

03

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Statement of inventorship and right to grant of a patent (Patents Form 7/77)

Request for preliminary examination and search (Patents Form 9/77)

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01

11. I/We request the grant of a patent on the basis of this application.

Signature

G Terry

Date 9 April 2002

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K M Pinder/G Terry 0161 721 1361/2

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PROCESS FOR CHEMICALLY PRODUCED TONERField of the invention

5 This invention relates to chemically produced toners for use in the formation of electrostatic images, their process of manufacture and to toner apparatus and components incorporating them. It further relates to any electroreprographic apparatus, component of the apparatus and consumable for use with the apparatus, which comprises such a toner, and to methods of manufacturing of such electroreprographic apparatus, components and consumables.

Related background art

10 Toners for development of an electrostatic image are conventionally produced by melt kneading of a pigment, resin and other toner ingredients, followed by pulverisation. Classification is then needed to generate an acceptably narrow particle size distribution.

15 Recently attention has been focussed on chemical routes to toners, where a suitable particle size is not attained by a milling process, which avoid the need for a classification step. By avoiding the classification step, higher yields can be attained, especially as the target particle size is reduced. Lower particle size toners are of considerable interest for a number of reasons, including better print resolution, lower pile height, greater yield from a toner cartridge, faster or lower temperature fusing, and lower paper curl.

20 Several routes to chemical toners have been exemplified. These include suspension polymerisation, solution-dispersion processes and aggregation routes. Aggregation processes offer several advantages including the generation of narrow particle size distributions, and the ability to make toners of different shape. The toner shape is particularly important in toner transfer from the organic photoconductor (OPC) to the substrate, and in cleaning of the OPC by a blade cleaner.

25 Several aggregation processes have been reported. US 4996127 (Nippon Carbide) reports a process in which black toner particles are grown by heating and stirring resin particles made by emulsion polymerisation with a dispersion of carbon black, where the resin contains acidic or basic polar groups. Numerous patents from Xerox (e.g. US 5418108) describe a flocculation process where particles stabilised by anionic surfactants are mixed with particles stabilised by cationic surfactants (or where a cationic surfactant is added to particles stabilised by an anionic surfactant). US 5066560 and US 4983488 (Hitachi Chemical Co.) describe emulsion polymerisation in the presence of a pigment, followed by coagulation with an inorganic salt, such as magnesium sulphate or aluminium chloride. The applicants' own patent applications WO 98/50828 and WO 99/50714, describe aggregation processes in which a surfactant used to stabilise the latex and

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pigment is converted by a pH change from an ionic to a non-ionic state, so initiating flocculation.

To form a permanent image on the substrate, it is necessary to fuse or fix the toner particles to the substrate. This is commonly achieved by passing the unfused image between two rollers, with at least one of the rollers heated. It is important that the toner does not adhere to the fuser rollers during the fixation process. Common failure modes include paper wrapping (where the paper follows the path of the roller) and offset (where the toner image is transferred to the fuser roller, and then back to a different part of the paper, or to another paper sheet). One solution to these problems is to apply a release fluid, e.g. a silicone oil, to the fuser rollers. However this has many disadvantages, in that the oil remains on the page after fusing, problems can be encountered in duplex (double-sided) printing, and the operator must periodically re-fill the oil dispenser. These problems have led to a demand for so-called "oil-less" fusion, in which a wax incorporated in the toner melts during contact of the toner with the heated fuser rollers. The molten wax acts as a release agent, and removes the need for application of the silicone oil.

There are many problems associated with the inclusion of wax in a toner. Wax present at the surface of the toner may affect the triboelectric charging and flow properties, and may reduce the storage stability of the toner by leading to toner blocking. Another problem frequently encountered is filming of the wax onto the OPC or the metering blade. If the wax is not well dispersed in the toner problems with transparency in colour toners can be found. With conventional toners, prepared by the extrusion/pulverisation route, it has only proved possible to introduce relatively small amounts of wax without encountering the above problems.

With colour toners, the demands on the toner to achieve oil-less release are much more severe than with monochrome printing. As typically four colours are used in full-colour printing, the mass of toner which can be deposited per unit area is much higher than with black printing. Print densities of up to around 2 mg/cm² may be encountered in colour printing, compared with about 0.4-0.7 mg/cm² in monochrome prints. As the layer thickness increases it becomes more difficult to melt the wax and obtain satisfactory release at acceptable fusion temperatures and speeds. Of course it is highly desirable to minimise the fusion temperature, as this results in lower energy consumption and a longer fuser lifetime. With colour printing it is also important that prints show high transparency. In addition it is necessary to be able to control the gloss level. Inclusion of waxes in colour toners can have detrimental effects on transparency, and can make it difficult to reach higher gloss levels.

The efficiency of wax melting can be increased by reducing the wax melting point. However this often leads to increased storage stability problems, and in more pronounced filming of the opc or metering blade. The domain size of the wax is also important, as this affects the release, storage stability and transparency of the toner.

The release properties of the toner can also be affected by the molecular weight distribution of the toner. Broader molecular weight distribution toners, which include a proportion of higher molecular weight (or alternatively cross-linked resin), generally show greater resistance to offset at higher fusion temperatures. However when large amounts of high molecular weight resins are included, the melt viscosity of the toner increases, which requires a higher fusion temperature to achieve fixation to the substrate and transparency.

Therefore the requirements for achieving an oil-less fusion colour system are severe. It is necessary to achieve a reasonably low fusion temperature, with an acceptably wide release temperature window, including with high print densities. The prints must show good transparency with controllable gloss. The toner must not show blocking under normal storage conditions, and must not lead to filming of the OPC or metering blade.

Several patents exemplify aggregation processes where a single latex, made by a one-stage emulsion polymerisation process, is aggregated with a wax dispersion. Examples where a system based on counterionic surfactants (i.e. an anionic and a cationic surfactant) is used include US 5994020 and US 5482812 (both to Xerox). Examples where an inorganic coagulant is used include US 5994020, US 6120967, US 6268103 and US 6268102 (all to Xerox). Mixed inorganic and organic coagulants are used in US 6190820 and US 6210853 (both to Xerox). US 4996127 (Nippon Carbide) exemplifies a process in which a latex containing an acidic-functional group is heated and stirred with a wax dispersion and carbon black to grow aggregate toner particles.

US 5928830 (Xerox) discloses a two stage emulsion polymerisation to make a core shell latex. The shell is made generally of higher molecular weight and/or Tg than the core. The latex is then mixed with pigment and flocculated through use of counterionic surfactants. Inclusion of wax is not exemplified.

US 5496676 (Xerox) discloses use of blends of different latexes with different molecular weight to increase the fusion latitude. Each latex is made by a single stage polymerisation. Toners were made by flocculating the mixed latexes with a pigment dispersion containing a counterionic surfactant. Inclusion of wax is not exemplified.

In US 5965316 (Xerox) encapsulated waxes are made by carrying out the emulsion polymerisation in the presence of a wax dispersion. These emulsion polymers containing wax are mixed with non wax containing latexes of similar molecular weight, and toners made using a counterionic flocculation route.

JP 2000-35690 and JP 2000-98654 describe aggregation processes where a non-ionically stabilised dispersion of an ester-type wax is aggregated with mixed polymer emulsions of different molecular weight.

US 5910389, US 6096465 and US 6214510 (Fuji Xerox) disclose blends of resins with different molecular weights, incorporating hydrocarbon waxes of melting point ~ 85°C. US 6251556 (Fuji Xerox) also discloses blends of resins, as well as a two stage

emulsion polymerisation to make a core shell latex. The only wax which is incorporated is a high melting point (160 °C) polypropylene wax.

Summary of the invention

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Therefore, obtaining a suitable toner, and a process for making it, which meets all these requirements is difficult and requires careful selection of the many possible components and parameters, each of which has constraints imposed on its physical and chemical properties by the final parameters of the system.

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According to the present invention there is provided a toner for developing an electrostatic image comprising a binder resin, a wax and a colorant, characterised in that the binder resin is prepared from at least one latex with monomodal molecular weight distribution and at least one latex with bimodal molecular weight distribution, and the wax has a melting point of between from 50°C to 150°C.

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We have found that by using an aggregation process with particular wax dispersions, it is possible to incorporate wax in relatively high amounts (e.g. about 5-15 wt%) without problems of blocking or filming, and without adverse effects on toner flow or tribocharge, or on print transparency. The wax is present in the toner in domains of mean diameter 2µm or less, preferably 1.5µm or less, and is not substantially present at the surface of the toner. The relatively high wax levels allow oil-less release even at high print densities, without requiring excessive amounts of high weight average molecular weight (M_w) resin. This allows fixation at low temperatures, and high transparency across a range of fusion temperatures. To achieve satisfactory oil-less release at high temperatures, it is necessary to have present polymer chains encompassing a wide range of molecular weights. This can be achieved either by mixing resin particles of widely different molecular weight, or by synthesising a latex containing a broad molecular weight distribution. A combination of both approaches can be used.

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Latexes may be made by polymerisation processes known in the art, preferably by emulsion polymerisation. The molecular weight can be controlled by use of a chain transfer agent (e.g. a mercaptan), by control of initiator concentration or by heating time. Latexes with a bimodal molecular weight distribution may be made using a two-stage polymerisation. Preferably a higher molecular weight polymer is made first, then in a second stage, a lower molecular weight polymer is made in the presence of the first polymer. As a result, a bimodal molecular weight distribution polymer is made containing both low and high molecular weight polymers. This is then mixed with a monomodal low molecular weight polymer. In a further aspect of the invention, three latexes can be used, where preferably at least two of these show bimodal molecular weight distributions. In a further preference, the second bimodal latex is of higher molecular weight than the first.

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Preferably the monomodal low molecular weight latex has a number average molecular weight of from 3000 to 10000, more preferably from 4000 to 10000. Where the

binder resin is prepared from one bimodal latex, the bimodal latex preferably has a weight average molecular weight of from 100,000 to 500,000, more preferably from 200,000 to 400,000. Where the binder resin is prepared from more than one bimodal latex, one bimodal latex may optionally have a weight average molecular weight from 500,000 to 1,000,000 or more.

The higher molecular weight polymers may also contain cross-linked material by inclusion of a multifunctional monomer (e.g. divinylbenzene or a multi-functional acrylate). It is preferred that the overall molecular weight distribution of the toner resin shows M_w/M_n of 3 or more, preferably 5 or more. The T_g of each resin is preferably from 30 to 100 °C, more preferably from 45 to 75 °C, most preferably from 50 to 70 °C. If the T_g is too low, the storage stability of the toner will be reduced. If the T_g is too high, the melt viscosity of the resin will be raised, which will increase the fixation temperature and the temperature required to achieve adequate transparency. It is preferred that all the polymers in the resin have a substantially similar T_g .

Optionally the latex dispersion further comprises an ionic surfactant; preferably the surfactant present on the dispersions contains a group which can be converted from an ionic to a non-ionic form by adjustment of pH. Preferred groups include carboxylic acids or tertiary amines. Preferably, the ionic surfactant has a charge of the same sign (anionic or cationic) as that of the surfactant used in the wax and pigment dispersions described below. Optionally a non-ionic surfactant may also be incorporated into the latex dispersion.

The wax should have a melting point (mpt) (as measured by the peak position by differential scanning calorimetry (dsc)) of from 50 to 150°C, preferably from 50 to 130°C, more preferably from 50 to 110 °C, especially from 65 to 85 °C. If the mpt is >150°C the release properties at lower temperatures are inferior, especially where high print densities are used. If the mpt is <50°C the storage stability of the toner will suffer, and the toner may be more prone to showing filming of the OPC or metering blade.

In a further embodiment of the invention, the wax is made as a dispersion in water, preferably stabilised with an ionic surfactant. The ionic surfactant is selected from the same classes as described above for the latex dispersion; preferably, the ionic surfactant has the same sign (anionic or cationic) as the surfactant used for the latex dispersion described above and the pigment dispersion described below. The mean volume particle size of the wax in the dispersion is preferably in the range from 100nm to 2 µm, more preferably from 200 to 800 nm, most preferably from 300 to 600 nm, and especially from 350 to 450 nm. The wax particle size is chosen such that an even and consistent incorporation into the toner is achieved. The wax should be present in the toner in domains, where the mean size of the domains is at most 2 µm, preferably 1.5 µm or less. If the mean size of the wax domains is > 2 µm, the transparency of the printed film may be reduced, and the storage stability may decrease.

The wax may comprise any conventionally used wax. Examples include hydrocarbon waxes (e.g. polyethylenes such as Polywax™ 400, 500, 600, 655, 725, 850, 1000, 2000 and 3000 from Baker Petrolite; paraffin waxes and waxes made from CO and H₂, especially Fischer-Tropsch waxes such as Paraflint™ C80 and H1 from Sasol; ester waxes, including natural waxes such as Carnauba and Montan waxes; amide waxes; and mixtures of these. Hydrocarbon waxes are preferred, especially Fischer-Tropsch and paraffin waxes. It is especially preferred to use a mixture of Fischer-Tropsch and Carnauba waxes, or a mixture of paraffin and Carnauba waxes.

The amount of wax incorporated in the toner is preferably from 1 to 30 wt% based on the total weight of toner, more preferably from 3 to 20 wt%, especially from 5 to 15 wt%. If the level of wax is too low, the release properties will be inadequate for oil-less fusion. Too high a level of wax will reduce storage stability and lead to filming problems. The distribution of the wax through the toner is also an important factor, it being preferred that wax is not present at the surface of the toner.

Preferably the colorant comprises a pigment. Any suitable pigment can be used, including black and magnetic pigments. For example carbon black, magnetite, copper phthalocyanine, quinacridones, xanthenes, mono- and dis-azo pigments, naphthols etc. Examples include Pigment Blue 15:3, Red 31, 57, 81, 122, 146, 147 or 184; Yellow 12, 13, 17, 74, 180 or 185. Preferably, the pigment is milled with an ionic surfactant, and optionally a non-ionic surfactant until the particle size is reduced, preferably to <300 nm, more preferably <100 nm. In full colour printing it is normal to use yellow, magenta, cyan and black toners. However it is possible to make specific toners for spot colour or custom colour applications. When the colorant is milled with an ionic surfactant, the surfactant is preferably selected from the same classes of surfactant described above for the binder resin and the wax; more preferably the surfactant has the same sign as both the surfactants used above.

The toner as described above may additionally optionally comprise a charge control agent (CCA); preferably the charge control agent has been milled with the colorant. Suitable charge control agents are preferably colourless, and include metal complexes, phenolic resins etc. Examples include Bontron™ E84, E88, E89 and F21 from Orient; Kayacharge N1, N3 and N4 from Nippon Kayaku; LR147 from Japan Carlit; TN-105 from Hodogaya. These can be milled in a similar manner to the pigment. Where the CCA is added externally, a suitable high-speed blender may be used, e.g. a Nara Hybridiser. Alternatively, the CCA may be added as part of the pre-flocculation mixture, preferably as a wet-cake.

In a further embodiment of the invention, there is provided a process for the manufacture of a toner according to the above which comprises the following steps:

- i. preparation of a latex dispersion;
- ii. preparation of a wax dispersion;
- iii. preparation of a colorant component;

- iv. mixing of the latex dispersion, wax dispersion and colorant component; and
- v. causing the mixture to flocculate.

The process may further comprise, prior to step iv, the additional step of preparation of a charge control agent component, which component is then incorporated in step iv by mixing.

The latex dispersion preferably comprises an ionic surfactant. More preferably the preparation of the latex dispersion comprises mixing together at least one latex with monomodal molecular weight distribution and at least one latex with bimodal molecular weight distribution. The preparation of the latex with bimodal molecular weight distribution preferably comprises the successive steps of formation of a polymer of high molecular weight distribution followed by formation of a polymer of low molecular weight distribution such that the resulting latex comprises composite particles comprising both the said low molecular weight polymer and the said high molecular weight polymer. The preparation of the wax dispersion in such a process preferably comprises the mixing together of the wax with an ionic surfactant. The preparation of the colorant component in such a process preferably comprises the milling together of the colorant with an ionic surfactant.

It is preferred that the dispersions of latex, pigment, charge control agent where present; and wax have the same sign charge on the surfactant. This enables individual components to be well mixed prior to flocculation. It is further preferred to use the same surfactant for each of the individual dispersions. The mixed dispersions are then flocculated. Any suitable method could be used, e.g. addition of an inorganic salt, an organic coagulant, or by heating and stirring. In the preferred method, the surfactant present on the dispersions contains a group which can be converted from an ionic to a non-ionic form by adjustment of pH. For example the surfactant contains a carboxylic acid group, and the dispersions are mixed at neutral to high pH. Flocculation is then effected by addition of an acid, which converts the surfactant from anionic to non-ionic. Alternatively the surfactant can be the acid salt of a tertiary amine, used at low pH. Flocculation is then effected by addition of a base which converts the surfactant from cationic to non-ionic form. The flocculation step is preferably carried out below the Tg of the polymer, but the mixed dispersions may be heated prior to flocculation.

The process as described above may optionally comprise heating the flocculated mixture to form loose aggregates of particle size from 3 to 20 μm ; in a further step of the same process, the aggregates are heated to induce coalescence, preferably to a temperature above the Tg of the latex or latex mixture.

Once the correct particle size is established, the aggregates are stabilised against further growth. This can be achieved, for example, by addition of further surfactant, and/or by a change in pH. The temperature is then raised to bring about coalescence of the particles within each aggregate. The cooled dispersion of coloured resin particles is then optionally washed to remove surfactant, and then dried.

The particles may then be blended with surface additives to improve the powder flow properties of the toner, or to tune the tribocharge properties. Typical surface additives include but are not limited to silica, metal oxides such as titania and alumina, polymeric beads (for example acrylic or fluoropolymer beads) and metal stearates (for example zinc stearate). Conducting additive particles may also be used, including those based on tin oxide (e.g. those containing antimony tin oxide or indium tin oxide). The metal oxide products, including silica, titania and alumina, may be made hydrophobic, e.g. by reaction with a silane and/or a silicone polymer. Examples of hydrophobising groups include alkyl halosilanes, aryl halosilanes, alkyl alkoxysilanes (e.g. butyl trimethoxysilane, iso-butyl trimethoxysilane and octyl trimethoxysilane), aryl alkoxysilanes, hexamethyldisilazane, dimethylpolysiloxane and octamethylcyclotetrasiloxane. Other hydrophobising groups include those containing amine or ammonium groups. Mixtures of hydrophobising groups can be used.

Examples of hydrophobic silicas include those commercially available from Nippon Aerosil, Degussa, Wacker-Chemie and Cabot Corporation. Specific examples include those made by reaction with dimethyldichlorosilane (e.g. Aerosil™ R972, R974 and R976 from Degussa); those made by reaction with dimethylpolysiloxane (e.g. Aerosil™ RY50, NY50, RY200, RY200S and R202 from Degussa); those made by reaction with hexamethyldisilazane (e.g. Aerosil™ RX50, NAX50, RX200, RX300, R812 and R812S from Degussa); those made by reaction with alkylsilanes (e.g. Aerosil™ R805 and R816 from Degussa) and those made by reaction with octamethylcyclotetrasiloxane (e.g. Aerosil™ R104 and R106 from Degussa).

The primary particle size of the silicas used is typically from 5 to 100nm, preferably from 7 to 40 nm. The BET surface area may be from 20 to 300 m²/g, preferably 30-150 m²/g. Combinations of silicas with different particle size and/or surface area may be used.

Where titania is used, it is preferred to use a grade which has been hydrophobised, e.g. by reaction with an alkylsilane and/or a silicone polymer. The titania may be crystalline or amorphous. Where crystalline it may consist of rutile or anatase structures, or mixtures of the two. Examples include grades T805 or NKT90 from Nippon Aerosil.

Hydrophilic or hydrophobic grades of alumina may be used. A preferred grade is Aluminium Oxide C from Degussa.

It is often preferred to use combinations of silica and titania (e.g. R972 or R812S with NKT90), or of silica, titania and alumina (e.g. R972 or R812S with NKT90 and Aluminium Oxide C). Polymer beads or zinc stearate may be used to improve the transfer efficiency or cleaning efficiency of the toners. Charge control agents may be added in the external formulation to modify the charge level or charging rate of the toners.

The total level of flow additives used may be from about 0.1 to about 10 wt%, preferably from about 0.5 to 5%. The additives may be added by blending with the toner, using, for example, a Henschel blender or a Nara Hybridiser.

The toner may be used as a mono-component or a dual component developer. In the latter case the toner is mixed with a suitable carrier bead.

In a further embodiment of the same invention, there is provided a process for manufacturing an electrophotographic apparatus and/or a component of the apparatus and/or a consumable for use with the apparatus, the process using a toner as described above.

In yet another aspect of the same invention, there is provided an electrophotographic apparatus, a component of the apparatus and/or a consumable for use with the apparatus, which comprises a toner as described above.

Examples

1. Latexes

1.1. Synthesis of Latex A-1

A low molecular weight resin was synthesised by emulsion polymerisation. The monomers used were styrene (82.5 wt%), 2-hydroxyethyl methacrylate (2.5 wt%) and acrylic ester monomers (15.0 wt%). Ammonium persulphate (0.5 wt% on monomers) was used as the initiator, and a mixture of thiol chain transfer agents (2.5 wt%) was used as chain transfer agents. The surfactant was AkypoTM RLM100 (available from Kao, 2.7 wt% on monomers). The emulsion had a particle size of 91nm, and a Tg midpoint (as measured by differential scanning calorimetry (dsc)) of 63°C. GPC analysis against polystyrene standards showed the resin to have Mn = 9,200, Mw = 25,000, Mw/Mn = 2.7. The solids content was 30 wt%.

1.2. Synthesis of Latex A-2

A low molecular weight resin was made as above, but the properties were: particle size 88nm, Tg midpoint (as measured by dsc) 62°C, Mn = 7,700, Mw = 19,000, Mw/Mn = 2.5. The solids content was 30 wt%.

1.3. Synthesis of Latex B

A bimodal molecular weight distribution latex was made by a two-stage polymerisation process, in which the higher molecular weight portion was made in the absence of chain transfer agent, and in which the molecular weight of the lower molecular weight portion was reduced by use of 2.5 wt% of mixed thiol chain transfer agents. Ammonium persulphate (0.5 wt% on monomers) was used as the initiator, and the surfactant was AkypoTM RLM100 (available from Kao, 3 wt% on monomers).

The monomer composition for the low molecular weight portion was the same as for Latexes A-1 and A-2 above. The overall monomer composition was styrene (73.85 wt%), 2-hydroxyethyl methacrylate (6.25 wt%) and acrylic ester monomers (19.9 wt%). The emulsion had a particle size of 80nm and a Tg midpoint (as measured by dsc) of 66°C. GPC analysis against polystyrene standards showed a bimodal molecular weight distribution with Mn = 35,000, Mw = 285,000, Mw/Mn = 8.1. The solids content was 40 wt%.

2. Pigment and CCA dispersion

A mixed dispersion of Pigment Red 122 (Hostaperm™ Pink E, Clariant) and Bontron E88 (Orient) was used. The pigment and CCA were milled in water using a bead mill, with Akypo™ RLM100 (Kao) and Solsperse™ 27000 (Avecia) as dispersants.

3. Wax dispersion

An aqueous wax dispersion was used which contained an 80:20 mixture of Paraflint™ C80 (Fischer-Tropsch wax from Sasol) and Carnauba wax. Akypo™ RLM 100 was used as the dispersant. The mean volume particle size of the wax was 0.4 µm, and the solids content 25 wt%.

4. Toner preparation

4.1 Comparative toner example

Latex A-2 (704.6g), the wax dispersion (90.6g), the mixed pigment and CCA dispersion (80.4g, containing 12.6g Pigment Red 122 and 5.0g of Bontron E88) and water (987g) were mixed and stirred in a glass vessel. The temperature was raised to 60°C. The mixed dispersions were pumped with a peristaltic pump from the vessel, through a flow cell equipped with an Ultra Turrax™ T50 high shear mixer operating at 10,000 rpm, and back into the vessel. As the material was circulating a 4% solution of sulphuric acid (150g) was added into the flow cell over 3 minutes. The flocculated mixture was then pumped back into the glass vessel and the temperature was then raised to 66°C. Stirring was continued for 3 hrs. A solution of 0.5N sodium hydroxide (194g) was then added to raise the pH to 7.3. The temperature was then raised to 91°C and stirring continued for a further 70 mins. The slurry was then cooled, and transferred to a Parr pressure vessel where it was heated with stirring for 5 minutes at 120°C. Coulter Counter™ analysis showed the mean volume particle size was 9.5µm and the final GSD was 1.30. The resultant magenta toner dispersion was filtered on a Buchner funnel, washed with water, dilute ammonia solution, water, dilute acid and finally water. The toner was then dried in an oven. A

portion of the toner was blended on a coffee grinder with 2 wt % of Aerosil™ R972 (Degussa) hydrophobic silica. The toner was then printed in a monocomponent monochrome printer which had been modified to remove the fuser, to allow printing of unfused images. The print density of a single pass print was found to be 0.7 mg/cm². The image was printed three times through the printer to generate an overall density of 2.1 mg/cm².

The images were then fused off-line using a QEA Fuser-Fixer equipped with a pair of heated oil-less fuser rollers. The fuser speed was set to 20ppm for images printed on paper, and 10ppm for images printed on transparencies for a overhead projector. For the prints on paper, hot offset was found to occur at 170°C. For the prints on transparencies hot offset was found at 150°C

4.2 Toner example 1

Latex A-1 (980 g), Latex B (183.8g) the wax dispersion (157.5 g), the mixed pigment and CCA dispersion (119.7g, containing 21.8 g Pigment Red 122 and 8.75g of Bontron E88) and water (1797g) were mixed and stirred in a glass vessel. The temperature was raised to 60°C. The mixed dispersions were pumped with a peristaltic pump from the vessel, through a flow cell equipped with a high shear mixer operating at 10,000 rpm, and back into the vessel. As the material was circulating a 4% solution of sulphuric acid (262.5g) was added into the flow cell over 3 minutes. The flocculated mixture was then pumped back into the glass vessel and the temperature was then raised to 66°C. Stirring was continued for 3 hrs. A solution of 0.5N sodium hydroxide (374g) was then added to raise the pH to 7.3. The temperature was then raised to 91°C and stirring continued for a further 70 mins. The slurry was then cooled, and transferred to a Parr pressure vessel where it was heated with stirring for 45 minutes at 120°C. Coulter Counter™ analysis showed the mean volume particle size was 8.9 µm and the final GSD was 1.28.

The resultant magenta toner dispersion was filtered on a Buchner funnel, washed with water, dilute ammonia solution, water, dilute acid and finally water. The toner was then dried in an oven.

A portion of the toner was blended on a coffee grinder with 2 wt % of Aerosil™ R972 (Degussa) hydrophobic silica. The toner was then printed in a monocomponent monochrome printer which had been modified to remove the fuser, to allow printing of unfused images. The print density of a single pass print was found to be 0.65 mg/cm². The image was printed three times through the printer to generate an overall density of 2.0 mg/cm².

The images were then fused off-line using a QEA Fuser-Fixer equipped with a pair of heated oil-less fuser rollers. The fuser speed was set to 20ppm for images printed on paper, and 10ppm for images printed on transparencies for a overhead projector. For the

prints on both paper and transparency, no hot offset was found to occur up to 190°C (the maximum fusion temperature studied)

CLAIMS

1. A toner for developing an electrostatic image comprising a binder resin, a wax and
5 a colorant, characterised in that the binder resin is prepared from at least one latex with
monomodal molecular weight distribution and at least one latex with bimodal molecular
weight distribution, and the wax has a melting point of between from 50°C to 150°C.
2. A toner according to Claim 1 wherein the monomodal latex has a number average
10 molecular weight of between from 3000 to 10000.
3. A toner according to either of claims 1 or 2 wherein the bimodal latex has a weight
average molecular weight of between from 100,000 to 500,000.
- 15 4. A toner according to any of the above claims wherein the latex further comprises
an ionic surfactant.
5. A toner according to any of the above claims wherein the wax is present in
domains of size $\leq 2\mu\text{m}$.
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6. A toner according to any of the above claims wherein the wax is incorporated from
a dispersion in water stabilised with an ionic surfactant.
7. A toner according to any of the above claims wherein the colorant comprises a
25 pigment.
8. A toner according to claim 7 wherein the pigment has been milled with an ionic
surfactant.
- 30 9. A toner according to claims 4 and 6 and 8 wherein the ionic surfactant comprised
in the latex has the same charge sign as the ionic surfactants comprised in the wax
dispersion and the colourant dispersion.
10. A toner according to any of the above claims which further comprises a charge
35 control agent.
11. A toner according to claim 10 wherein the charge control agent has been milled
with the colorant.

12. A process for the manufacture of a toner according to any of the above claims which comprises the following steps:

- i. preparation of a latex dispersion;
- ii. preparation of a wax dispersion;
- iii. preparation of a colorant component;
- iv. mixing of the latex dispersion, wax dispersion and colorant component; and
- v. causing the mixture to flocculate.

13. A process according to claim 12 wherein the latex dispersion comprises an ionic surfactant.

14. A process according to claim 12 or claim 13 wherein the preparation of the latex dispersion comprises mixing together at least one latex with monomodal molecular weight distribution and at least one latex with bimodal molecular weight distribution.

15. A process according to claim 14 wherein the preparation of the latex with bimodal molecular weight distribution comprises the successive steps of formation of a polymer of high molecular weight distribution followed by formation of a polymer of low molecular weight distribution such that the resulting latex comprises composite particles comprising both the said low molecular weight polymer and the said high molecular weight polymer.

16. A process according to any of claims 12 to 15 which, prior to step iv, further comprises the step of preparation of a charge control agent component, which component is then incorporated in step iv by mixing.

17. A process according to any of claims 12 to 16 wherein the preparation of the wax dispersion comprises the mixing together of the wax with an ionic surfactant.

18. A process according to any of claims 12 to 17 wherein the preparation of the colorant component comprises the milling together of the colorant with an ionic surfactant.

19. A process according to any of claims 12 to 18 wherein the flocculated mixture is heated to form loose aggregates of particle size from 3 to 20 μm .

20. A process according to claim 19 wherein the aggregates are heated to a temperature above the T_g of the latex to induce coalescence.

21. A process for the manufacture of an electrophotographic apparatus and/or a component of the apparatus and/or a consumable for use with the apparatus, the process using a toner as claimed in any of claims 1 to 11.

22. An electroreprographic apparatus, a component of the apparatus and/or a consumable for use with the apparatus, which comprises a toner as claimed in any of claims 1 to 11.

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23. A compound, composition, electroreprographic apparatus and method for making any of the aforementioned, which is substantially as described herein with reference to the Examples.

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